Year 3 Annual/Final Report

SYNTHESIS AND CHARACTERIZATION OF POLYMERIC MATERIALS

Contract NOOO14-90-C-2269

GC-TR-94-2303

GEO-CENTERS, INC.

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This report is a summary of GEO-CENTERS' research efforts in support of Naval Research Laboratory under Contract N00014-90-2269 entitled, "Synthesis and Characterization of Polymeric Materials". The period of performance was from 28 September 1990 to 15 August 1994. The majority of the work was carried out at NRL and NRL's Chesapeake Bay Detachment using Chemistry Division, Acoustics Division, Materials Science & Technology Division, and NRL-CBD Support facilities, and in collaboratorion with NRL staff and scientists. Additional work was carried out at the U.S. Army RD&E Center in Natick, MA. The various research projects involved research in three main areas -- evaluation of sonar dome damage, polymer synthesis and evaluation, and bio-degradable material for packaging.

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INTRODUCTION

This report is a summary of GEO-CENTERS' research efforts for the Naval Research Laboratory (NRL) under contract number N00014-90-C-2269, entitled "Synthesis and Evaluation of Polymeric Materials". The period of performance was from 28 September 1990 through 15 August 1994. The majority of the work was carried out at NRL and NRL's Chesapeake Bay Detachment (CBD) using Chemistry Division, Acoustics Division, Materials Science & Technology Division, and NRL-CBD Support facilities, and in collaboration with NRL staff scientists. Additional work was carried out at the U.S. Army RD&E Center in Natick, MA.

The work has resulted in several publications in scientific journals, presentations at various scientific meetings, and patent citations. Documentation of these works is provided in the Bibliography Section of this report; each citation is referred to in the text by brackets [ ] surrounding a bibliography reference number.

The various research projects in which GEO-CENTERS has been involved under this contract are divisible into three main groups: evaluation of sonar dome damage, polymer synthesis and evaluation, and bio-degradable packaging material.

The sonar dome projects have involved fleet support functions as well as research. GEO-CENTERS, INC. has provided the interpretation and analysis of radiographic inspection data in support of a routine fleet sonar dome inspection program. We have also conducted failure analyses of individual domes. Research projects have focused upon nondestructive evaluation methods, sonar dome dynamic mechanical analysis, and new materials characterization. In addition, database management support for the sonar dome effort have been provided. Discussions of these efforts are in the first part of the technical report.

In the area of synthesis and evaluation, GEO-CENTERS has conducted research on the synthesis of novel materials, such as fluoropolymers, epoxies, conductive polymers, phthalonitrile resins, elastomeric blends, and modified nylons. The tasks have also involved the characterization and evaluation of these materials with respect to their mechanical, rheological, chemical, and electrical properties. In addition, the characterization methods have been investigated and improved upon where applicable. Discussions of these accomplishments are provided in the second part of the technical report.

In addition to these areas of research, GEO-CENTERS has provided technical support to various non-research projects in the Chemistry Division, such as the Networking Task. These efforts are presented as the third part of this report.

TECHNICAL REPORT

1.0 EVALUATION OF SONAR DOME DAMAGE

1.1 Background

Sonar Dome Rubber Windows (SDRW) and Sonar Rubber Domes (SRD) are installed on Navy surface combatants to provide a protective window for the transmission of sonar signals. These structures provide a hydrodynamic fairing around the sonar transducer arrays for the purpose of eliminating turbulence and system self-noise. They also protect the sonar system from the actions of the sea and collisions with debris.

Sonar domes are currently fabricated from a steel cord reinforced rubber composite material. The SDRW is a large, bow-mounted structure which is fitted to eleven classes of cruisers, destroyers, and frigates. The SRDs consist of two types, which are directly associated to specific sonar systems--AN/SQS-56 and AN/SQQ-23. The AN/SQS-56 SRD is a 273-inch-long keel-mounted dome installed on *Perry* class frigates, while the AN-SQQ-23 SRD is a 400-inch-long keel dome installed on four classes of cruisers and frigates which were phased out of service in U.S. fleets. In the following summary, we will refer to all of the structures as "sonar domes". SDRW or SRD will be used when specifically applicable.

Sonar domes have a history of rupturing during service. Studies performed by NRL have demonstrated that the SDRW failures are due to corrosion fatigue failure of the steel reinforcement cords in the splice region of the composite structure. The SRD failures are not as well understood; however, corrosion fatigue has been identified during SRD failure analyses.

X-ray radiography has been used to detect incipient corrosion fatigue damage. An inspection program has been set up with the goal of maintaining an up-to-date evaluation of the entire anti-submarine warfare fleet's sonar domes. Since replacement domes are also subject to the same problems, the need to inspect, monitor, and repair or replace them still remains. Radiographic inspection has been routinely used as a basis for determining these options. In addition, the accumulated radiographic data have contributed to the failure analysis effort by revealing patterns of damage distribution and to statistical models which have enabled us to guide NAVSEA in their long range planning for sonar dome construction. The inspection and analysis methods developed in response to the SDRW failure problem have since become applicable for use on similar problems with the smaller keel domes. However, the inspection of these structures requires removal of the dome in drydock. This prompted NAVSEA to investigate new non-

destructive methods of SRD inspection. GEO-CENTERS has responded by collaborating with NRL on a major effort toward achieving a one-sided nondestructive inspection technology for sonar domes.

GEO-CENTERS' support of the sonar dome program has been in the areas of radiographic inspection, improved methods for nondestructive evaluation, database management, failure analysis, and new materials testing and development. The following paragraphs summarize these efforts.

1.2 Radiographic Inspection

GEO-CENTERS' role in the radiographic inspection program has involved the interpretation of radiographs provided by the Navy's inspection contractors, development and maintenance of standards for the radiographic data, as well as the criteria for determining the action to be taken upon completion of the radiographic evaluation.

As the sonar dome radiographs arrive, they are processed immediately. Any damage or pertinent features are located, measured, and diagrammed either as a hand sketch or as a computer generated image. The appropriate recommendation for action to be taken is based on the standardized criteria devised at NRL. The results are communicated verbally to the NRL task manager and/or others designated by NRL (typically NAVSEA 91W4D). The data are also entered into the appropriate database, and a report is generated for distribution.

1.2.1 Inspection Results

During the final period of performance, GEO-CENTERS has evaluated 56 radiographic sonar dome inspections, and 249 over the course of the contract. For each inspection, a report was written detailing our findings, including illustrations of the inspection area and damage locations. The recommended action is determined by a set of well defined and established criteria. The inspection reports are used in determining the action to be taken on a given SDRW or SRD. Accumulations of these reports are provided as appendices in a summary memorandum to NRL. These memoranda are then distributed to NAVSEA as NRL letter reports. Fourteen such letter reports have been generated during the contractual period [41,44,46,51-54,56,59-61,68,69,74], along with two individual reports regarding the inspection of domes on foreign vessels [71,75].

1.3 Improved Nondestructive Evaluation (NDE) Methods

1.3.1 X-ray Backscatter Tomography (XBT)

Keel mounted Sonar Rubber Domes (SRDs) on more than fifty Navy surface combatants are subject to unexpected rupture at sea. Repair can only be accomplished in dry dock. Ruptured domes render their sonar system inoperative until emergency dry docking can be scheduled, therefore, deferring the required maintenance slated for another ship. The mechanisms of stress corrosion, corrosion fatigue, and tensile failure have been implicated. X-ray radiography is used to manage a similar failure problem with the larger bow mounted SDRWs. Both drydock and pierside (underwater) techniques are used. SRD radiography, however, requires access to the dome interior. Since keel domes have no airlock, access is limited to drydock availabilities. Furthermore, the AN/SQS-56 SRD, installed on FFG-7 class frigates, must be removed from the hull for inspection.

Technical Objective/Expected Payoffs

A pierside SRD inspection capability which would considerably improve the effectiveness of the SRD inspection program is being developed. Payoffs are expected in the following areas:

Costs per SRD inspection will be lowered, due to the elimination of dry-docking ($1M per occurrence) and removal requirements. Furthermore, eliminating emergency SRD repair will eliminate "bumping" other combatant dry dock schedules and the snow-ball cost impacts to the Navy.

Inspections will be more frequent, with timing based upon technical considerations and pierside availability rather than drydock availability. This will allow the identification of high risk SRDs and monitoring of lesser damaged SRDs. The expected decrease in failures-at-sea will result in increased operational readiness.
Identification of the damaged SRD population will facilitate planning for procurement to maintain spare inventories.

Inspection data will contribute to the ongoing SRD failure analysis and corrective action effort.

Technical Background and Approach

SRDs, also known as keel domes, rupture due to the failure of the steel cord elements of the steel/rubber composite dome wall. Preliminary failure analysis efforts suggest a failure mechanism similar to that seen in the larger bow mounted SDRWs. The failure scenario involves water intrusion via cracks, migration via voids or cord wicking, corrosion of cords exposed to the water, and fatigue of weakened cords during flexure of the dome wall.

The same methods developed for SDRW inspection are also currently used to inspect SRDs. Using standard industrial radiography procedures, film is placed on the inside surface of the SRD and the area is irradiated from the outside to form an x-ray transmission image. The limitation of being able to access only SRDs removed from the hull in drydock has handicapped the inspection program.

In order to apply the lessons learned in the SDRW program to SRDs and to achieve similar success in managing this problem, it will be necessary to inspect all of the SRDs in service and to accumulate periodic inspection data. The current procedure is inadequate to achieve these goals. A pierside inspection methodology is needed. The technology must be sufficiently sensitive to detect internal SRD damage and be capable of operating from the exterior of the submerged dome.

X-ray backscatter tomography (XBT) is an one-sided x-ray technology which provides image data representing the composition of a planar slice within the inspected object. The process is based upon the Compton scattering interaction between incident x-rays and electrons in the material. Each volume element, or voxel, in the slice is interrogated by a narrow x-ray beam and collimated scatter detectors. Both the location of the voxel and its scatter signal are acquired and used to form an image. Multiple slices can be acquired to represent the whole volume to be inspected. Unlike the better known computed tomography (CT), XBT requires access only to the x-ray source side of the object.

Both resolution and required dwell time per voxel are determined by the x-ray beam and detector aperture sizes. Because of this trade-off, systems providing practical inspection times have lower resolution than provided by radiography. However, they have the advantage of three dimensional resolution. Since the beam and scatter signal are both modulated by material outside the voxel being measured, XBT has usually been applied to homogeneous materials which are either thin or of low density where undesirable effects are minimized. Numerous applications of XBT have been proposed in the literature, and a few have been developed to the demonstration phase. However, this technology has never been widely implemented due to the above mentioned

trade-offs and to the availability of the well developed CT technology. Some XBT problems are amenable to other solutions, such as ultrasound. Only recently has there been a suitable x-ray scanner on the market, obviating the need for costly development of a one-of-a-kind scanner.

Our goal has been to develop and field an XBT inspection system suitable for the in-situ inspection of AN/SQS-56 SRDs. To minimize development costs, the system is based on a commercially available x-ray backscatter scanner, Philips ComScan.

The criteria for successful inspection has been the detection of failed steel cords in the outer longitudinal ply and runs of broken cords greater than 1 inch in the second longitudinal ply. Attempts have also been made to resolve damage to the deeper overlapped longitudinal structures. XBT evaluations were verified by comparison with radiographic inspection data and by physical dissection of the inspected SRDs.

There are three task areas in the developmental effort:

Mechanical Prototype - The XBT system consists of a number of sub-systems: a power conditioner, power supplies, high voltage generator, x-ray system controller, computers, three CRT monitors, heat exchanger for x-ray coolant, data acquisition boards, servo controls, and a remote x-ray scanner head with cables and coolant hoses. This equipment was to be installed in an insulated transportable container that would have lighting, heating, air-conditioning, sound absorbing panels, a shock mounted floor, water-tight doors, and overhead trolley crane for deployment of the scanner. Hardware was to be developed for attaching the scanner to the ship's hull and for indexing the scanner through a sequence of positions on the SRD surface. An underwater scanner enclosure with 20 meter umbilical to the surface was also in the development plan.

Methodology and Data Acquisition - This involves the inspection of SRDs removed for cause and those that become available in drydock. Inspection coverage requirements was to be determined and scanner parameters will be optimized. Data would be compared with radiography results. Underwater inspections would ultimately be demonstrated. Data acquisition, storage and transmission subsystems were to be developed and modified as required.

Data Analysis - New computer reconstruction methods would be needed to meet inspection sensitivity objectives. This requires the characterization of system performance. A

computer model of the process would be developed to aid in the evaluation of reconstruction strategies. Data visualization methods would be evaluated and implemented.

Summary of Work Completed During the Period of Performance

Fabrication of the shipping/utilization container (SUC) was completed, and the XBT system components were installed for field operation. A light-weight manipulator was designed and fabricated to provide 5-axis control of the scanner for sonar dome inspection trials. A trailer was outfitted to provide workshop and storage space. The SUC has been located on an existing concrete pad at the Chesapeake Beach site where space is also available for removed sonar domes inspection trials.

Operating procedures were written and approved by the Navy Radiation Affairs Safety Office [65]. XBT inspections were conducted of damaged SRDs removed from the *USS CLARK* (FFG-11), *USS FAHRION* (FFG-22), *USS BOONE* (FFG-28) and *USS FLATLEY* (FFG-28). These domes had been radiographed and found to contain a number of typical damage sites. The known damaged areas were inspected using the XBT system with various scanning speeds, apertures, and orientations. The data produced were compared with the transmission radiographs. The results demonstrated the ability of XBT to detect keel band damage but not the deeper bead band damage. Also, in one case, keel band damage was undetected unless the dome was loaded with water. This indicated that broken cord ends must be separated a certain distance to be resolved. Pressurization of domes attached to ship was proposed to ensure such separation. This effect was subsequently demonstrated during drydock trials.

The XBT system was sent to both Navy and private shipyards to perform in-situ SRD inspections during drydock availabilities of the *USS NICHOLAS* (FFG-47) [70], *USS HAWES* (FFG-53) [72] and *USS ELROD* (FFG-55) [76]. Shipyard support and logistics needs were fine-tuned and documented. The XBT inspections were successful, resulting in recommendations to replace all three SRDs. Two were radiographed upon removal and the XBT results verified. In one case the XBT results were considered superior to those of radiography. As a result of these demonstrations NRL has recommended that NAVSEA accelerate the transition of the drydock XBT inspection capability to Fleet use. This option will now be provided for FFG-7 class drydock availabilities when dome removal is not planned.

The XBT system has also been made available for investigating other applications of interest to the government. For example, studies were completed for heat damaged graphite epoxy

composite (Navy) [24] and wall thickness determination of corroded low-level radioactive waste barrels (Department of Energy) [84]. Other such collaborations are currently underway.

With regard to the problem of resolving deeper structure, the volumetric data has been found to contain artifacts of the XBT process. The most damaging artifact to interpretation is caused by attenuation of both primary and scattered x-rays by overlying steel cords. Commercially available software has been evaluated and selected for processing and enhancing the XBT image data. Software has also been developed for automating the enhancement and cataloging of the voluminous XBT inspection data sets. Work is progressing in collaboration with an NRL/ONR postdoc on the development of computer reconstruction and image processing methods to increase the effective depth of XBT inspection and improve interpretation. Both a Monte Carlo simulation and an analytic model of the XBT process have been completed towards this goal. Studies of various aspects of the problem have resulted in several publications and presentations [2,3,4,5,22,23,27,29], and a patent disclosure (by an ONR Postdoctoral Fellow).

The current emphasis is on development of the underwater capability. After discussions with Navy underwater maintenance experts, designs were modified to incorporate their suggestions [64]. An enclosure to house the XBT scanner has been fabricated and tested at a 40 ft depth [73]. A 65 ft. (20 meter) underwater umbilical has been installed with attendant modifications to system electronics. An underwater hydraulic positioning system has been designed; fabrication will be completed in FY 95. The underwater system demonstration will be delayed until this hardware is available.

Progress on this project has been presented at ONR technical reviews for two successive years [21,25].

Additional publications, presentations, and reports of and about this work were prepared during the period of performance [1,6,20,26,28,40,62,63].

Planned Work

The following plan is proposed towards the completion of the XBT project during a follow-on effort:

The drydock inspection capability will be transitioned into the Fleet. Drydock inspections will be conducted as requested.

The underwater positioning system will be fabricated and tested in drydock. Underwater XBT inspection will be demonstrated. Additional pierside inspections will be conducted depending on ship availability.

The image reconstruction development work will be completed and implemented.

Both drydock and pierside inspections will be conducted as requested by the Fleet. XBT system components will be modified and finalized as indicated during inspection operations.

A final report on the XBT system will be prepared, as well as a dome inspection manual and system specifications and technical manuals.

1.3.2 Other Methods

Although emphasis and resources have been placed on the development of XBT as a new methods of non-destructive evaluation, other methods have been proposed and investigated [57] as candidates for one-sided inspections of SRDs. In comparison to XBT, these other methods promise to be less expensive and easier to implement, although their resolutions have proven to be lower than that of XBT. These methods may be utilized as inexpensive, preliminary screening techniques for identifying the most seriously damaged SRDs. Based on the results from such a preliminary screening, a more detailed evaluation using XBT could be implemented. This scenario was addressed in a publication entitled "One-Sided NDE for Sonar Rubber Domes" [1].

1.3.3 Image Processing of Radiographic Data

Our most current work in this area is discussed in Section 1.3.1. Prior investigations carried out during the period of performance involved the use of artificial neural networks for pattern classification applications. In the first year annual report, we discussed the application of neural networks in sonar dome cord profile analysis [7,30]. Our efforts progressed toward applying neural nets in the quantitative measurement of object features from radioscopic data [31]. Using a computer simulation of the x-ray transmission data, we have generated network training and testing data sets for a variety of flaw measurement problems. It has been shown that the neural network's tolerance to variation in object location or orientation makes this technique ideal for automated inspection applications, which would otherwise require accurate data registration. A

feed-forward, back-propagation network configuration and various representations of the profile data have been used.

Image processing techniques, and the application of neural networks for the processing of SDRW and SRD radiographic and XBT data, promise to improve the non-destructive evaluation of these structures in the future.

1.4 Database Management

Developing and maintaining databases are important to many aspects of the sonar dome program. Under previous contracts, GEO-CENTERS developed databases for SDRW and SRD tasks. Currently, records are maintained on every SDRW/SRD installed or newly manufactured. Complete histories of each SDRW/SRD (installation, radiographic inspections, repairs, removal) are available. Inventories of SDRW and SRD spares are maintained, and records of domes on ships decommissioned to foreign navies are also tracked. In addition, the development of a database application used in conjunction with NAVSEA's optical storage medium for documents helps interface system users to that database [58].

The SDRW/SRD databases have provided a library of information for statistical analysis. Previously, analysis of SDRW damage distributions from inspection data had helped effect design changes and enabled us to develop recommendation criteria and inspection schedules. This analytical capability has also allowed for the occasional review of these criteria [77]. These analyses have fostered the ability to reasonably predict a SDRW's life expectancy under varying conditions, and allow NAVSEA to better project annual budgets for SDRW purchases and maintain fleet readiness. Analysis of SRD damage distributions have also been done [50]. The results have allowed us to limit the region of interest when conducting investigations of new methods for nondestructive testing and when performing analysis on failed SRDs.

Finally, results from the analysis of SDRW inspection data permitted an increase in the recommended radiographic inspection interval for new and undamaged six-ply rubber windows. In collaboration with an NRL reliability statistician, we determined that a sub-population of SDRWs merit being called "improved", and led to an increase in the inspection interval in these cases. Our progress was documented in four NRL letter reports [45,47,48,55].

During this reporting period, GEO-CENTERS has continued to maintain all previously created databases and to develop new database applications for SDRW and SRD as required by

the expanding needs of the sonar dome program. With exception to the AN/SQQ-23 database which handles inactive vessels, all database structures continually undergo program editing and overhaul. In short, maintaining our capability to provide NAVSEA with up-to-date and prompt analyses of sonar dome data has been the underlying goal of this effort.

1.5 SDRW/SRD Corrective Action Programs

1.5.1 Background

The Navy's Corrective Action Program (CAP) was initiated in response to the problem of SDRW failures at sea. The program was originally comprised of a team of scientists and engineers proceeding on a number of parallel courses, united by the common goal of solving this problem. Through various methods of analysis the primary cause of failure was determined, and improvements were made by developing a routine radiographic inspection program. Eventually, changes in the SDRW design were made based on the analyses. This resulted in increasing the projected service life of these structures. GEO-CENTERS' participation involved providing technical support to the CAP team, and in advising the NRL task manager on technical issues.

The SDRW CAP's past success in discovering the chief mechanism of failure in SDRWs, and the subsequent engineering changes taken to minimize its effects, have not diminished the program's efforts toward continued overall improvement. Previous recommendations of the CAP have led to a new monolithic SDRW design, which must now be evaluated using dynamic-mechanical analytical methods. GEO-CENTERS has continued to support this effort as well. In particular, we have been closely involved with the instrumentation of the new SDRW, and will provide support for the testing phase. This will be discussed further in Section 1.5.3.

Although laboratory analyses provided an understanding of problems with SDRWs, due in part to its complexity, a complete understanding of the failure mechanism in SRDs has yet to be realized. Consequently, though new design changes have increased the projected service life of SDRWs, the same claim cannot be made for SRDs. As a result, the SRD CAP is now progressing along two parallel courses: (1) dynamic response and structural analysis of SRDs, which includes failure analysis and deflection measurement (Sections 1.5.2 and 1.5.4, respectively); and (2) new design analysis and materials testing (Section 1.5.5).

1.5.2 Failure Analysis

Sonar domes fail when the amount of sustained structural damage severely compromises the load carrying capability of the dome. At that point, rupture is likely to occur, allowing the dome to lose its internal pressure. This is known as a failure at sea. If a sonar dome is inspected prior to rupture, and the damage is beyond what the criteria allows for a dome in service, it is removed. This is regarded as a failure by inspection (X-ray). To be assured that the best pre-conditions exist for laboratory analysis, only the failure-by-inspection sonar domes are analyzed. During the period of performance, failure analysis reports on two SRDs were completed [66,67], and one (*Ex-USS Clark*) is in the interim stage.

In the former two analyses, an investigation of the bead neck region yielded results that led to an engineering change (mold-ring modification, ECP #6037-31) in the manufacturing process. In addition, we conducted a feasibility study for effecting a structural bead neck repair [42,43,49]. In the latter investigation, a goal has been set to determine the location of water migration pathways which are believed to have provided the means for the onset of corrosion in the SRD.

1.5.3 Cord Load Measurements

Background

Information about the forces experienced by sonar domes at sea is needed for design feedback, and can be used for engineering changes. It is a necessity, therefore, that the acquisition of the cord-load data be completed for every newly designed sonar dome prototype produced. This provides NAVSEA with an assessment of the dynamic reactions of the structure well before full implementation in the fleet. Through the study of a sonar dome's dynamic character while at-sea, we can provide data to current models for quantitative structural analysis. Additionally, the new design may be compared to earlier prototypes in a qualitative manner. Ultimately, this method of testing enables us to work toward the goals of the CAP.

The first six-ply spliceless (monolithic) SDRW was fabricated by B.F. Goodrich (BFG) as layup 252, bearing the serial number 1A. During construction, load cells were placed within the structural plies to be used in the measurement of loads in critical areas after installation on a ship. GEO-CENTERS has provided technical and logistical support to this task. This has involved gauge placement and calibration, cabling, and data processing. It has also involved the coordination with planned acoustic deflection measurements, taken concurrently.

The first phase of this project required the calibration of all the load cells and instruments prior to curing of the SDRW. Initial calibration was performed by the manufacturer under GEO-CENTERS and NRL observation. A post-cure load (pressurization) test was then performed, where all the cells were taken to an operational status. The second phase involved testing the cured dome at various pressures, and checking the equipment setup and computer algorithms. The third phase will encompass all preparations needed to eventually install the systems on board a Navy vessel.

Load Cell Calibration/Measurement

The SDRW manufacturer installed 85 calibrated load cells on SDRW-1A. These load cells consist of a 36x2x2 mm brass billet with four active gages in a full bridge configuration. Two of the active gages were mounted for maximum strain; the other two were mounted for Poisson strain. Each load cell was supplied with a factory assigned serial number, gage factor, offset value, and an initial load calibration.

All load cell calibrations were done at B.F. Goodrich in Jacksonville, Florida. During the calibration and subsequent testing, GEO-CENTERS provided technical support. Pre-cure base line readings were taken at 75 °F after SDRW layout completion. The excitation voltage (Vex) was 4 VDC. Three readings were taken per cell for verification. All 85 cells were tested by measuring the resistance between the cell's strain gages (a properly functioning strain gage should read approximately 350 ohms). The cells were tested before and after installation into the plies. Three cells failed after an induced voltage was applied, and were replaced. Due to production time constraints, pre-cure loads were not taken. Analysis of the data revealed that the cells were linear within the expected load range. Baseline readings were then taken for all the load cells.

The second phase of the project involved pressurizing the SDRW and using a prototype equipment setup. Readings were taken at 0 psig with the dome empty, and at 16.5 psig, 35.6 psig, and 56.0 psig with the dome full of water. The load cell cables installed by BFG proved to be highly susceptible to damage and water intrusion, and some cells were found to be malfunctioning. These cables would eventually be replaced after we found a suitable alternative, and administered the appropriate tests.

In the third phase, GEO-CENTERS will provide logistical and technical support to the continued dome preparation, installation of devices and equipment, and in the acquisition of data for the project.

1.5.4 SRD Acoustic Deflection Project

Through the CAP, NAVSEA has an ongoing effort to determine the cause and to prevent the future occurrences of sonar dome failures. An important part of the program is the at-sea measurement of sonar dome structural response to ship motions, internal pressurization changes, and sea state, under operational conditions. These data can then be used to validate finite element models necessary for assessing the probable effectiveness of future design changes for failure prevention.

Acoustic deflection measurement is a method for monitoring discrete movement of positions on the SRD's surface, while making correlative measurements of pertinent environmental conditions. An array of hydrophones is attached to the inner surface of the dome, whereupon their acoustic signals are continuously converted to three-dimensional positions during the sea-trial.

GEO-CENTERS was a participant during the preparation, data acquisition, and data processing phases of the project. Initially we collaborated on the design, construction, positioning, and installation of a sonar transducer array mock-up for the project. We also provided logistical and technical support in the measurement, placement, and installation of the numerous ultrasonic transducers to be used in this project. We developed a mapping system for the interior surface of the SRD, which enabled us to translate a given surface location to the standardized x-y-z coordinate system used by the SRD manufacturer and other Navy activities. We then assisted in the system installation, and fully participated in the actual at-sea experiment.

Data processing included the following: the arrangement of original data into a spreadsheet format and applying correction factors; the conversion of the original x-y-z data into surface normal vectors; and the calculation of the deflection range, minimum, and maximum surface normal deflections.

Our collaborative effort, with scientists from NRL and SFA, was documented in an NRL Memorandum Report [79].

1.5.5 New Materials/Design - Testing and Analysis

As part of the Corrective Action Program, GEO-CENTERS is often called upon to test and/or evaluate the materials and processes used in sonar dome manufacture. In 1992, the current sonar dome manufacturer proposed a new composite design, termed RHO-COR®, as a replacement

for the now used AN/SQS-56 SRD. The design utilizes a sandwich-style architecture, which would maintain a rigid structure, and fiber reinforced plastic composites as new material components. The NRL task manager solicited comments on the new design from GEO-CENTERS and other CAP participants, and the manufacturer later followed with a proposal to construct a prototype. Since that time, we have collaborated with NRL to enter a preliminary testing phase, whereupon design evaluation and materials testing was initiated.

During the period of performance, GEO-CENTERS has assumed a coordinating role in the preliminary testing and evaluation phase of the proposed sonar dome design. We have advised the NRL task manager on pertinent and appropriate tests for the RHO-COR® design, and have appropriated samples of the composite sandwich from the manufacturer for examination. A program for preliminary testing has been initiated, utilizing facilities at NRL, and at the NRL detachment at Key West, Florida. The test outline, and a list of applicable documents, is provided as Appendices A & B.

2.0 POLYMER SYNTHESIS AND EVALUATION

The Materials Chemistry Branch is responsible for the development and characterization of new polymeric materials for Navy applications. GEO-CENTERS' support of the branch's efforts has been broad based involving the synthesis and characterization of many varied materials. The main focus has been in developing synthetic polymers that emulate the elastic linkages found in natural polypeptides, and polymers that yield enhanced dielectric properties, though other areas of interest are discussed here as well. Characterization has involved several diagnostic methods including different types of thermal analyses, scanning electron microscopy (SEM), transmission electron microscopy (TEM), nuclear magnetic resonance (NMR), electron spin resonance (ESR) and infrared and ultraviolet spectroscopic analyses. The methods and techniques of characterization have been investigated to meet the increasing requirements for better precision in the polymeric research. In addition, we have used mechanical, rheologic, and dielectric tests when appropriate. The specific project areas are discussed in the summaries which follow.

2.1 Fluorinated Polymers

The highly polar nature of the carbon-fluorine bond has been used to provide improved properties as compared to the hydrogen-containing or other halogen-containing analogs. Fluorine-containing epoxies or acrylics generally exhibit resistance to water penetration, chemical reaction,

and environmental degradation; they also show differing degrees of surface tension, friction coefficient, optical clarity, refractive index, vapor transmission rate, and electromagnetic radiation resistance.

Initially, this task included efforts by GEO-CENTERS to maximize the hydrophobicity of polymer systems without compromising the structural characteristics which are necessary in the development of polymeric materials. The program progressed toward the design and synthesis of heavily fluorinated epoxy and acrylic resins to produce compounds with dielectric constants among the lowest in the literature. This effort produced an entirely new class of polymers, resulting in the awarding of a U.S. patent [81], and subsequent patent disclosures in the U.S. and Europe [81,82]. A new process for preparation and synthesis, along with descriptions of the structure-property relationships are contained in five publications [13,14,16,17,18], and in works presented at various scientific meetings [35,36,37,38,39].

We have also been invited to submit samples of our polymers for publishing in Sadtler Standard Spectra, an infrared (IR) spectra reference book, and to contribute further by submitting manuscripts for the inclusion as chapters in books on fluoropolymers [83].

The emphasis of this task has been more recently directed toward the development of these fluoropolymers, by optimizing the reaction conditions to produce compounds of high molecular weight. We have also worked toward lowering impurity concentrations and achieving higher yields. These compounds have been prepared in various forms, such as thin films and blocks, to demonstrate processability and to facilitate characterization and evaluation for scaled-up production.

2.2 L-Proline Modified Nylons

The conformational constraints that L-proline imposes upon natural polypeptides has been recognized as the most significant factor in the occurrence of ß bends and pleats. The amino group of this most unusual natural α-amino acid is secondary and contained in a five-membered ring structure. One consequence of this, the peptide linkage formed with the amine group lacks an available hydrogen atom for hydrogen bonding. The implications for the materials properties, as opposed to the biological properties, of natural and synthetic polyamides containing L-proline are most interesting. A synthetic effort was initiated by first defining the minimal structure of synthetic polymer for which L-proline will control the elastic properties.

Two publications, the second of which is in revision, are the current results of this effort [12,15]. In addition, we presented results of this work at the American Chemical Society meeting in 1991 [34].

2.3 Elastomers and Blend Characterization

Polymer blends have been heavily investigated in recent years. Our early work in the area of rubber blends reported a miscibility due to specific interactions in a polychloroprene/ epoxidized polyisoprene mixture [33].

Recently ^{129}Xe NMR has been used to study polymer blends. The inertness of xenon and the dependence of its chemical shift on the local environment make it an attractive probe of morphology. It is particularly advantageous for blends whose components exhibit proximal glass transition temperatures, or specific morphologies that produce minute changes in the thermal response of a compound. Standard thermal analysis techniques, such as calorimetry, are somewhat insensitive to these characteristics [32]. Through the use of ^{129}Xe NMR, it has been possible to gain a resolution advantage over conventional methods. In addition, xenon is soluble in many polymers, with the atoms residing in the free volume. Since the chemical shift of ^{129}Xe is proportional to the density of its environment, it provides a measure of the free volume in the polymer. Below the glass transition temperature, segmental motion of the polymer chains is suppressed, so that Xe atoms are trapped in different sites. The distribution in the local free volume produces variations in the chemical shift, resulting in an inhomogeneously broadened ^{129}Xe NMR line. Rapid diffusion of the polymeric components collapses the inhomogeneously broadened resonance into a narrow line; the observed chemical shift is an average over the inhomogeneously broadened line. GEO-CENTERS participated in the characterization and subsequent evaluation of the blends for this work, and co-authored a publication on the subject [8].

2.4 Conductive Polymers

Our approach to developing electrically conducting organic materials that have high stability in aggressive environments is based on the synthesis of polymers with extended π-electron delocalization. The prepolymers are soluble and meltable, and completely conjugated with reactive end groups that are capable of being polymerized into conjugated/aromatic linking groups to form the polymer structure. Because of the extended conjugation, these materials are intrinsically

conducting without doping. We have studied the mechanical properties and thermal behavior of such a conducting polymer, demonstrating that the high temperatures used to introduce conductivity have not resulted in charred materials lacking mechanical integrity. To the contrary, we have seen improved tensile strength, as well as high oxidative and thermal stability. A paper on this topic was accepted for publication in the Journal of Applied Polymer Science [10].

2.5 Phthalonitrile Resins

Organic polymers that are both thermally and oxidatively stable above 315°C are in demand as matrix materials for advanced composite applications, as adhesives for high temperature materials, and as weight-reducing replacement materials for metals. A new class of phthalonitrile-based polymers with excellent thermoxidative properties has been under investigation for application in these areas. The phthalonitrile monomers are readily converted to crosslinked thermosetting polymers in the presence of thermally stable aromatic diamines. The polymerization reaction occurs through the terminal phthalonitrile units which are interconnected by aromatic dioxy linkages, yielding heterocyclic crosslinked materials. Shaped components are easily processed by heating the polymerization mixture above its melting point or glass transition until gelation occurs. The prepolymers formed from these monomers are soluble in common solvents and indefinitely stable at room temperature.

During the period of performance, the tensile failure and fracture properties of the phthalonitrile polymer following exposure to elevated temperature and oxidative conditions were detailed in a publication in Polymer Communications [11].

2.6 Nylon Webbings

The synthetic webbings in an air drop parachute system connect the parachute lines with the various pieces of military or industrial equipment involved in the drop. In order to predict the performance of such a system, one must study the interaction of the system with the surrounding air during deployment. A mathematical model was developed by Allied Signal Corporation to predict the system's behavior and estimate the magnitude of strain energy and stresses in the webbing sling.

To assist Allied in evaluating webbings of two types of nylons wound at two separate facilities, we developed a test utilizing NRL facilities. A summary report, prepared by Allied Signal using our data, outlines the results of this effort [78].

3.0 BIO-DEGRADABLE PACKAGING MATERIAL

An area of biotechnological research concerns biodegradable packaging for use aboard Navy ships or by U.S. Marine Corps land forces. This work involves the exploitation of starch-based packaging to develop packaging materials that will convert organic mass to carbon dioxide, water, and methane. Since successful biodegradation will require microbial-mediated mineralization, significant effort is focused on learning degradation rates of candidate materials and their mechanical properties.

The foregoing requirements pose a unique challenge to the biological and materials sciences. They will require basic and applied research in a wide variety of biotechnology programs.

The work performed during the period of performance of this contract addressed the following projects:

- the development of bio-degradable packaging film, especially for shipboard use in accord with the restrictions of the MARPOL treaty on waste disposal at sea;

- the development of advanced, environmentally compatible materials from renewable resources.

Two publications based on the support of these projects were generated [19, 80]. A detailed summary of these efforts is also contained in Appendix C, which is separately bound.

4.0 FOLLOW-ON WORK AND MISCELLANEOUS TECHNICAL SUPPORT

4.1 Deuterated Polymers

In continuation of support efforts initiated on the previous contract with the Materials Chemistry Branch, GEO-CENTERS participated on the current contract in a variety of follow-on research projects, such as, the development and evaluation of deuterated polymers. During the period of performance, a publication was submitted to the *Journal of Polymer Science* based on a collaborative effort with NRL on examinating deuterated polymers [9].

4.2 Wind-Tunnel Sample Collection System

During a previous contract with NRL, we provided hardware and software to support a data acquisition project. The system collected target residues from within a wind tunnel environment. We designed and fabricated a portable computer-based system to collect atmospheric samples within a wind tunnel.

During field testing of the at White Sands, NM, damage to the electronics was sustained. During the period of performance on the current contract, we repaired the damaged electronics and upgraded the system to a more robust design. Software and the system documentation were also updated to accommodate the design modifications.

4.3 Ethernet Networking Task

The *Internet* is a vast network of networks that offers users access to volumes of information on a wide array of topics. More than a means for communication, the Internet provides computer-to-computer connectivity between schools, libraries, universities, laboratories, government agencies, public and private organizations, military activities, and the general public. Coupled with a TCP/IP (Transport Control Protocol/Internet Protocol) compliant interface, an Internet connection affords the user the capability to view information, and obtain and transfer extremely large files in very little time, without incidence of error. This is vital to scientists and engineers needing the information, and facilitates communications for collaborative research between distant parties.

During the period of performance, GEO-CENTERS was requested to render assistance in the implementation of a Chemistry-Division-wide effort to connect PC computer users to the NRL wide-area network, and thus the *Internet*. For our part, this was to involve the Materials Chemistry Branch in particular, but also would include providing assistance directly to the division superintendent. As it turned out, no one in the division had the expertise to assure connectivity; thus some branches, including Materials Chemistry, were without anyone to assume the role of administering the planned effort. GEO-CENTERS' role in the Materials Chemistry Branch was thereby revised to manage this task, and to additionally assist at the division level.

Until recently, access to the NRL network was not provided to most desktop PCs. By 1993, however, NRL connectivity plans included making provisions for all PCs in all buildings at the lab. By 1994, a new fiber-optic network "backbone" was being implemented in the Chemistry building, and the network was optimized to accept a higher volume of users. It was during this time that we organized the connectivity effort.

GEO-CENTERS worked closely with technicians and programmers from NRL's Networking Group to insure that all interested Chemistry PC-users were satisfactorily connected to the NRL network. This involved the procurement of more than thirty cable transceivers and computer ethernet cards, and their installation, as well as the routing of nearly two-thousand feet of ethernet cable. In addition, we obtained all the necessary software from the Networking Group and other remote sources, and subsequently programmed all the PCs with the capability to gain access to the NRL network and numerous internet resources.

Currently, Materials Chemistry network users have the means to access any computer worldwide that accepts anonymous log-in via *Telnet* or *FTP* (File Transfer Protocol). Users preferring menu-driven access have been provided with the software connecting them to *Infonet*, the NRL library's Internet accessing system. This service provides direct access to various libraries and databases, publication search facilities, the NRL supply inventory, Internet news groups, and electronically published texts. We have also provided users with software to enhance the electronic-mail facilities at NRL.

We have continually provided software upgrades on all of these systems, as the NRL network backbone has changed dramatically since our initial installations. In addition, we have provided technical support in all situations when the systems malfunction, and have continued to solicit assistance from the NRL Networking Group when necessary. This has often required editing and rewriting program files to accommodate the needs of an ever-changing system of network users.

In the near future, those NRL scientists in control of project funds will have access, through the network, to the Chemistry Division's finance database. Program managers will be more in control of the disbursement of funds, and have a more accurate account of funding levels than at present.

4.4 Miscellaneous Research Support Tasks

GEO-CENTERS frequently gives technical support to the Materials Chemistry Branch pertaining to test equipment maintenance, computer hardware and software support, training, materials testing, and test preparation. The following is a list of some of the tasks that have been accomplished during this period of performance:

Equipment Maintenance/Personnel Training--We have performed routine maintenance on various test devices and analytical equipment -- such as, dynamic rheomteters, GC/MS, DSC/TGA, DSC/TMA, and MTS servo-hydraulic cyclic-loading tester. This has involved routine assessment of the equipment's performance, system calibration, installation of options and upgrades, and system refits. New users are regularly trained and supervised on these systems.

Computer Hardware/Software Upgrades--In the Materials Chemistry Branch, GEO-CENTERS has performed numerous computer hardware installations, upgrades, and repairs -- such as, PC upgrade from 286 to 386 to 486, and HP to PC UNIX-based upgrades. Software upgrades have been installed -- such as, DOS, Windows, and WordPerfect -- and new database programs were written in Rbase and Dbase to allow effective communication between various locations at NRL.

Materials Testing/Sample Preparation--GEO-CENTERS has been involved with short-term testing for various projects within the Materials Chemistry Branch. During the first year, we completed both destructive and non-destructive testing and an evaluation of sonar dome contract samples for NAVSEA. During this period of performance, we have prepared polymer samples for testing by another Naval facility. Since laser surface oblation experiments were to be carried out on these samples, a preparation technique was developed to ensure that a class "A" quality surface resulted. Several different sizes of low and high density polyethylenes with varying melt indexes were prepared.

APPENDIX A:

Outline for Testing of the RHO-COR® Composite Structure

Appendix A: OUTLINE FOR TESTING OF THE RHO-COR® COMPOSITE STRUCTURE

I. MECHANICAL CHARACTERIZATION OF FRP ATTACHMENT

1. Bearing Strength.
   a) Adapter plate area.
   b) Threaded insert area.
2. Tension testing using determined FEA values.
3. Flexure testing.
   a) Flexural properties.
   b) Creep testing.
   c) Fatigue testing.
4. Shear testing.
   a) Shear properties.
   b) Shear fatigue.
5. Impact testing.

II. MECHANICAL CHARACTERIZATION OF RHO-COR COMPOSITE

1. Compression testing of sandwich.
2. Tension testing using determined FEA values.
   a) Parallel to the facing plane.
   b) Normal to the facing plane (will also check adhesive strength)
3. Flexure testing.
   a) Flexural properties.
   b) Creep testing.
   c) Fatigue testing.
4. Shear testing.
   a) Shear properties.
   b) Shear fatigue.
5. Impact Testing.

III. ENVIRONMENTAL CHARACTERIZATION OF FRP ATTACHMENT

1. Water absorption of FRP.
2. Blistering effects from surface anomalies.
    a) Scratches
    b) Cracks and localized damage.
3. Aging effects.
4. Characterization of bearing strength, tensile strength and flexure in sea/polluted water.
5. Crevice corrosion of the attachment plate at the FRP interface.

IV. ENVIRONMENTAL CHARACTERIZATION OF RHO-COR COMPOSITE

1. Water absorption of core material.
2. Aging of core material.
3. Characterization of tensile strength and flexure in sea/polluted water environment.

APPENDIX B:

Documents Applicable to RHO COR® Testing Listed in Appendix A

Appendix B: DOCUMENTS APPLICABLE TO RHO-COR® TESTING LISTED IN APPENDIX A

Definitions will be according to ASTM C 274-68 (88)

Standard Practice for Determining and Reporting Dynamic Mechanical Properties Of Plastics -- D 4065-92

I.

Supplement: Glass-Fiber-Reinforced Polyester Plastic Panels -- D 3841-92

Supplement: Determining and Reporting Dynamic Mechanical Properties of Plastics -- D 4065-92

Supplement: Test Method for Rockwell Hardness of Plastics -- D 785-89

1. a) Bearing Strength of Plastics -- D 953-92
   b) Bearing Strength of Plastics -- D 953-92
2. Tensile Properties of Plastics -- D 638-91
3. a) Flexural Properties of Unreinforced and Reinforced Plastics and Insulating Materials -- D 790-92
   b) Flexural Creep -- D 790-92 or FTMS 406, 1063
   c) Basic Fatigue Test using FEA and sea trial data. Test Method for Flexural Fatigue of Plastics by Constant Amplitude of Force -- D 671-90
4. a) In-Plane Shear Strength of Reinforced Plastics -- D 3846-79
   b) Modified for Fatigue: In-Plane Shear Strength of Reinforced Plastics -- D 3846-79
5. Modified for RHO-COR/Plastic Dynamic Tear Test. Test Method for Dynamic Tear Testing of Metallic Materials -- E 604-88

II.

1. Edgewise Compressive Strength of Flat Sandwich Constructions -- C 364-61 (88)
   Supplement: Flatwise Compressive Strength of Sandwich Cores -- C 365-57 (88) [modulus of elasticity]
2. a) Standard tensile test. Test Methods for Rockwell Hardness of Plastics -- D 785-89
   b) Tensile Strength of Flat Sandwich Constructions in Flatwise Plane -- C 297-61 (88)
3. a) Flexural Properties of Flat Sandwich Constructions -- C 393-62 (88)
   b) Flexure Creep of Sandwich Constructions -- C 480-62 (88)
   c) Basic Fatigue Test using FEA and sea trial data. NRL prescribed test -- Modified D 790-92 and D 671-92

4. a) Shear Properties in Flatwise Plane of Flat Sandwich Construction or Sandwich Core -- C 273-61(88)
   b) Shear Fatigue of Sandwich Core Materials -- C394-62 (88)
5. Dynamic Tear Test -- E 604-current

III.

1. Water absorption of plastics. Test Method for Water Absorption of Plastics -- D 570-81(88) -- and/or Practice for Determination of Weight and Shape Changes of Plastics Under Accelerated Service Conditions -- D 756-78 (83)
2. a) Modified D 570-81(88)//D 756 (83)
   b) Modified D 570-81(88)//D 756 (83)
3. Accelerated Weathering Test -- FTMS 460, 6023
4. All of section I testing modified to evaluate specimen with sea/polluted water contamination. Resistance of Plastics to Chemical Reagents -- D 543-87

IV.

1. Water Absorption of Core Materials for Structural Sandwich Constructions. C 272-91
2. Laboratory Aging of Sandwich Constructions. C 481-62 (88)
3. Applicable test under section II modified to evaluate specimen with sea/polluted water contamination. Resistance of Plastics to Chemical Reagents D 543-87

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